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# INVESTIGATION OF CRITICAL DEFECTS IN ABLATIVE HEAT SHIELD SYSTEMS

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#### ABSTRACT

An experimental program was conducted under NASA Contract NAS1-10289 to determine the effects of fabrication-induced defects on the performance of ablative heat shield materials. Critical defects were identified and methods of their detection during inspection were established. Steps to streamline the fabrication and inspection processes were then postulated in order to lower the mass production costs of these material composites in the event that they were to be used on a Space Shuttle Orbiter.

#### INTRODUCTION

Ablative materials have been proven as heat shields in space flight. Because reliable, low cost heat shield materials are required for Space Shuttle vehicles, ablators are now considered to be confident backup candidates to the current baseline shuttle thermal protection system. Prime considerations in this application are: mission function, producibility and inspectability. We have recently completed a program for NASA-Langley Research Center (Contract NAS1-10289<sup>1</sup>) on the study of the effects of anomalies in elastomeric ablative materials. This work is the basis for data presented in this paper.

Our study of the effects of ablator anomalies was divided into five tasks:

- Task I Identification, nondestructive evaluation and characterization of potentially critical defects;
- Task II Application of nondestructive evaluation techniques to ablative panels which had been fabricated by four different manufacturing processes;
- Task III Evaluation of various fabrication-induced defects on ablator performance in a simulated space shuttle reentry environment;
- Task IV Characterization of various fabrication-induced defects on Space Shuttle environments prior to reentry;

Task V Updating current state-of-the-art nondestructive evaluation techniques for ablative heat shield certification.

The overall objectives of this approach were to establish quantitative engineering acceptance criteria and low cost production and assurance methods based on performance requirements. Since ablator materials function in both active and passive performance, our study involved both physical and physical-chemical properties evaluation.

#### GROUND RULES (TASK I)

In establishing the basis for our study involving the effects of fabrication flaws, it was necessary to select from the many aspects of missions, vehicle locations, environments, possible defects and design configurations a baseline approach which could be adequately investigated within the scope of this program. In addition, a proper definition of the performance expected was vital in that it created guidelines against which the reactions could be measured.

# Definition of Criticality

A critical defect was established as a perturbation of the ablative system that affected critical properties to the extent that the system did not meet basic performance requirements. In particular, these depended on the deterioration of the composites structural and/or thermal capabilities due to flaws. Flaws introduced at one point in the material's history could fully develop to a critical defect later in the sequence of the mission environments and thereby affect performance in a phase such as reentry.

#### Investigation Point

Thermal protection requirements were based on the bottom centerline region of a Shuttle Orbiter on a logistic resupply mission.

#### Origin of Flaws

All variances were assumed to be introduced before completion of the panel assembly. Furthermore, assuming the raw materials meet specific acceptance criteria, the majority of flaws were initiated during the subcomponent fabrication or final assembly.

#### Assembly Configurations

The heat shield assembly construction used in the first half of this effort consisted of a full depth phenolic-glass honeycomb filled with an elastomeric ablator (MG-36) and bonded to a fiber-glass backface sheet, see Figure 1. A honeycomb subpanel TPS

design was selected for the second half of the studies. In addition, the ablative fill was altered to a mixture identified as SS-41, see Figure 2. These changes were enacted as a result of Task I, II and III evaluations to: (1) provide support for the ablator in the presence of positive and negative normal airloads, (2) establish a more reproducible baseline material composite, and (3) introduce some latitude to the materials and combinations considered.

#### INITIAL CAUSE AND EFFECT TESTING (TASK III)

Using the MG-36 design, flaw-characterized specimens were investigated. The procedure involved the determination of the reactions of defects in a reentry heating environment immediately after fabrication, except for two wedge panels which were exposed to other environments before and after heating. An ancillary phase investigated the mechanical properties of defective samples over a range of temperatures. Simultaneous studies involving production and assurance methods evaluation were also initiated. The latter are discussed later.

# Reactions to Entry Heating Only

Cylinders of ablator material MG-36, with defects, were exposed to end splash heating in the MMA Plasma Arc Facility. These were machined from large billets in the form of 5-in. diameter, 2-in. thick slugs.

<u>Defects</u>--The 68 slug specimens concentrated individually on variances in density (15 to 18 pcf), poor priming of the filler's honeycomb reinforcement (off B-staging, overloading), internal voids (up to 25% of the volume), formulation (fiber substitution and omission), overcuring, and undercuring.

Entry Heating Exposure and Results—Steady state cold wall heating rates ( $\dot{q}_{cw}$ ) of 23 or 55 BTU/ft²—sec (at stream enthalpies ( $\rm H_r$ ) of 3800 or 6400 BTU/lb) were exposed in the plasma arc facility for 1200 or 900 seconds, respectively. The following observations were noted: (1) low density was not critical with respect to excessive backface temperature until it dropped to about 80% of the nominal density, (2) an excess amount of the ablator core wet-coat resin created a maximum backface temperature violation because of an increase in net thermal conductance through the material, and (3) voids initiated in the ablator at fabrication were not critical until they reduced the net density of the medium to the 80% value. Omission of silica fibers was not found to be critical. Also, large variations in cure temperature, pressure and time did not appear to be critical in this test set.

# Reactions to Entire Mission Spectrum

Two panels (8 x 16 x 2-in.) were investigated to initially evaluate TPS response to a typical sequence of Shuttle environments. One panel was defect-free while the other had a variety of defects located about the panel planform.

<u>Defects</u>--The flaws incorporated were as follows: all the defects examined in the first entry heating set, plus inclusions, non-homogeneity, surface voids, undercut core, crushed core, face sheet disbond from the ablator, and face sheet delaminations.

Exposures—Those entry and non-entry environments which were believed to be the most likely to precipitate critical defects from manufacturing flaws during their period of influence were simulated individually on the panels. The environments were, in order: ascent acoustics (up to 154 db), hot/cold vacuum (+300°F (72 hour) and -320°F (48 hour) at  $10^{-5}$  torr), entry heating (panel inclined at 20 degrees,  $\dot{q}_{cw}$  (avg) = 18 BTU/ft<sup>2</sup>-sec, H = 11,700 BTU/lb, for 1,000 seconds), and descent acoustics (154 db max for 4 minutes).

Observations--No significant degradation of the test panel appearances or performances was noted until after the entry heating exposure. There, local areas of unsupported char cracked into a random pattern, with little difference noted between the regions of crushed core and undercut core. Char retention elsewhere was not noticeably affected by these localized core defects or by the surface discontinuities created by ablator material removal. A facility failure destroyed the control panel at about the midpoint in the run; no adverse conditions were noted in the undestroyed fragments. The descent acoustics spectrum, subsequently imposed on the defect panel, created additional char losses in the areas of crushed core and new failures in the regions of undercut core and undercured material.

# Mechanical Properties Evaluation

Tensile tests of representative coupons of control and defective ablator billets were conducted at temperatures ranging from  $-150^{\circ}\mathrm{F}$  to  $300^{\circ}\mathrm{F}$  to ascertain any notable changes in physical properties which could affect the performance of the material. The specimens were conventional 7-in., necked-down configurations.

<u>Defects</u>--The potential defects included in this strength investigation involved wet-coat variations, density variations, altered cures, and material omissions.

Observations -- Ultimate tensile strengths decreased for wet-coat variations, off densities, and altered cures. The same was true for ultimate elongation percentages. The data also indicated the

absence of any increase in tensile modulus for any of the flaws.

#### FINAL CAUSE AND EFFECT TESTING (TASK IV)

This task concentrated on the determination of the criticality of manufacturing flaws in ablative thermal protection systems relative to many environments and influences. It would have been desirable that all conceivable manufacturing variations be tested in all possible environments throughout the service life of a Shuttle ablative TPS. However, the matrix of potential environment-flaw combinations had to be reduced to a practical program which hopefully encompassed the significantly critical items.

As previously explained, this portion of the program had the ablator fill material change from MG-36 to SS-41 and the substrate from a single sheet to a honeycomb subpanel. The 27 composite panels used were 22 x 17.5 x 2-in. prior to subsequent subdivision for the later tests.

# Defects

There were ten basic flaw characteristics examined in the final phase, with two variations of each. (The balance of the panels (7) were controls.) Several of the defects selected were among those previously examined. The new flaws, also utilizing two panels each were: horizontal delaminations just below the surface, high filler moisture contents, broken honeycomb ribbons and broken honeycomb nodes. A summary is presented in Figure 3.

# Environments

Several additional environments and progressive quality inspections were added to the list of operations performed on investigation panels. The complete sequence associated with each wave of panels (with a wave consisting of a group of eight) is graphically illustrated in Figure 4.

All three waves were exposed to a single 24-hour, 98% relative humidity cycle at  $140^{\circ}\mathrm{F}$ . They then received an average overall ascent acoustic excitation of 163 db for one minute. This was followed by uniaxial flexure (in the weak direction) such that the strain on the outer face of the ablator was the equivalent of 1/2%. Thermal vacuum cycling followed, with one extended period (40 hours) at  $+200^{\circ}\mathrm{F}$  and five cycles between room temperature and  $-150^{\circ}\mathrm{F}$ , all at  $10^{-5}$  torr.

The panels were then subdivided, with the major portion  $(8 \times 12\text{-in.})$  converting to an inclined specimen for entry heating simulation (at 20 degrees) in our Plasma Arc Facility. Seven beams per panel were machined  $(8 \times 4\text{-in.})$  for failure testing (in the unpyrolized state) in four-point flexural assemblies.

The latter beams were cut along either the strong or the weak panel ribbon direction.

The nominal plasma arc conditions were:  $\dot{q}_{cw} = 13~BTU/ft^2$ -sec,  $H_r = 8550~BTU/lb$ , time = 1100 seconds, and static pressure of 15 torr. These exposures were followed by descent acoustic environments (151 overall db level for two minutes). Centerline sectioning of each specimen was the final operation, terminating in visual examinations of the ultimate influences of the flaws.

# **Observations**

A complete reiteration of the data and rationales utilized to highlight the critical defects finally designated is beyond the scope of this presentation. Figure 5 summarizes all the adverse comments made with respect to the intentional flaw, the defect symptoms noted, and the environments in which they were detected.

Based on the limits of this study, therefore, it would appear that the fabrication flaws which must be avoided focus on: ablator undercuring and overcuring, ablator low density, improper B-staging of the reinforcement honeycomb core's wet-coat priming, and any form of undercut or crushed core. These flaws have, in this study, displayed adverse combinations of excessive backface temperature, internal char fracturing, surface deterioration, and material losses. The crucial period begins at entry and continues to the safe return of the vehicle.

PRODUCTION AND ASSURANCE METHODS EVALUATION

#### Quality Assurance Orientation

Initial work with the baseline MG-36 material consisted of the evaluation of fabrication process variations and evaluation of potential nondestructive evaluation methods. The baseline process consisted of bonding a fiberglass backface sheet to the honeycomb core, packing the ablator from the open cell side, during the billet and mechanical machining of the excess "head" material to a constant thickness. Numerous evaluation methods were investigated.

X-radiography was determined to be sensitive to honeycomb core anomalies, bulk density variations, voids and contamination. A critical reaction to honeycomb "wet coat" and resin component composition was identified. This sensitivity overshadowed all other effects, thus limiting the usefulness of the technique.

Neutron Radiography was affected by honeycomb core anomalies, bulk density variations, voids and contamination. It was less sensitive than x-radiography to wet coat and resin variations

and was also less influenced by other anomalies.

Radiographic Image Processing provided additional discrimination and quantitative readout of x-ray images. The technique extended the potential for radiographic techniques but was influenced by the same phenomena which is inherent to the radiographic processes.

<u>Indentation Hardness (Shore A)</u> was sensitive to ablator cure, density and moisture content, with cure demonstrating the greatest indications. The technique was somewhat variable. Variations were attributed to resin-rich areas near the honeycomb cell wall and to the large indentation produced.

<u>Microwave</u> examinations reacted to ablator thickness, moisture content, resin content, density, cure, packing variations and wet coat variations. Its sensitivity to a number of anomalies made identification of a critical defect difficult.

Thermal (Infrared) scanner techniques were influenced by voids and density variations but were less sensitive than x-radiography.

Sonic and Ultrasonic techniques could determine unbonds and debonds from the facesheet side but could not penetrate the ablator material.

Holographic Interferometry provided some detection of voids and unbonds, but results were difficult to repeat.

These nondestructive evaluation techniques were applied to characterize test specimens used in Task III assessment of defect criticality in a simulated space shuttle reentry environment.

# Fabrication Process Evaluation (Task II)

X-radiography, indentation hardness and sonic techniques were selected for Task II evaluation of panels produced by four different processes. These processes were specified to result in identical end item panels. Nondestructive evaluation demonstrated variations in both processes and materials. Dissection of a section of each panel verified nondestructive evaluation results. This effort resulted in a change in both the material and the fabrication process to the SS-41 designation for the remainder of the program. Changes included slight composition variation, a change in wet coat from a silicone to a phenolic resin, a change to packing the ablator mix in honeycomb which was open from both sides, curing in billet form and secondarily bonding to a honeycomb substrate support panel. These changes considerably improved producibility and inspectability.

# Assurance Methods Refinement (Task V)

X-ray, indentation hardness, ultrasonic and holographic techniques were selected to characterize the SS-41 ablator material and for further refinement.

<u>X-radiometric Gaging</u> parameters were determined for monitor of variations in ablator density. One-half pound per cubic foot changes in ablator density were determined at energies ranging from 65 to 75 kilovolts. Separate calibration curves were necessary for each ablator thickness. X-ray (or gamma ray) gaging was determined to be applicable to production of the SS-41 material and could be automated for low cost production control.

X-ray evaluation for soundness was also determined to be applicable to the SS-41 material and could be automated. In-motion radiographic techniques using both film and electronic monitoring were evaluated. A penetrometer sensitivity of approximately 30% was determined for monitor by electronic (video) techniques. Video image analysis was determined to be feasible to direct, automatic readout of the resultant image for voids and density variations.

Indentation Hardness was monitored by a modified Shore "D" durometer. Modifications consisted of changing the indenter foot to a flat disc configuration and reducing the spring load on the indenter. These modifications enabled monitoring of the resilience of the ablator without variations due to crushing. No permanent mark was made in the ablator by the indenter. Greater consistence of measurements was obtained by this technique, but it was more sensitive to changes in panel thickness than was the Shore A unit used in previous evaluation. The modification was concluded to be useful due to greater consistency of results and elimination of the indenter mark.

Sonic/Ultrasonic evaluation of secondary bonding of the ablator to its subpanel was made using a sonic resonator (Model 101C). Unbond and debonds larger than one inch in diameter were detected from the subpanel side. Considerable variation in resonance response was experienced within a panel and successive evaluations of panels after environmental exposure. Known unbonds and debonds were consistently detected but variations in instrument response made inspection difficult. Sample panels were dissected and the variations were concluded to be due to differential absorption of the adhesive into the ablator material. Variations after environmental exposures were attributed to changes in modulus due to cure and/or absorbed moisture changes. Further refinement would be necessary for application of the sonic technique to full scale production.

<u>Holographic</u> evaluation results were negative on the SS-41 material. Loss in sensitivity was attributed to the increase in material permeability which prevented vacuum stressing of the material.

X-ray, Shore A and Shore D modified indentation hardness and sonic resonator methods were applied to evaluation and monitor of Task IV panels after various exposures. All techniques were determined to be applicable and meaningful in evaluating critical defects identified.

#### CONCLUSIONS

From this program we conclude that:

- 1. Elastomeric ablators are "very forgiving materials." No catastrophic defects analogous to cracks in metals were identified. Critical defects, i.e., defects which result in out-of-tolerance performance, are the result of out-of-tolerance variations in fabrication.
- 2. Critical defects identified were: a) ablator undercure and overcure; b) low ablator density; c) poor bonding of the ablator to the honeycomb support; d) undercut or damaged honeycomb core; e) large internal voids.
- State-of-the-art inspection methods will detect all critical defects identified with the exception of poor bonding to the honeycomb core. Bonding is readily assured by process control.
- 4. Elastomeric ablators are proven heat shield materials and could be applicable to space shuttle missions for primary or for "off-the-shelf" backup utilization in leading edge or total surface vehicle protection with incorporation of future streamlining of production and inspection methods.

# REFERENCES

- Miller, C. C. and Rummel, W. D., "Study of Critical Defects in Ablative Heat Shield Systems for the Space Shuttle," NASA CR Document to be published, 1973.
- Thompson, R. L., Rummel, W. D., and Driver, W. E., "Study of Critical Defects in Ablative Heat Shield Systems for the Space Shuttle, Tasks I, II and III", NASA CR-2010, April, 1972.

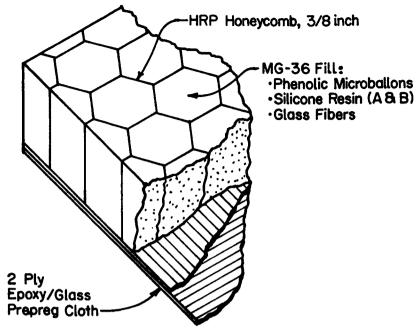


Figure 1 - Baseline Configuration - MG-36

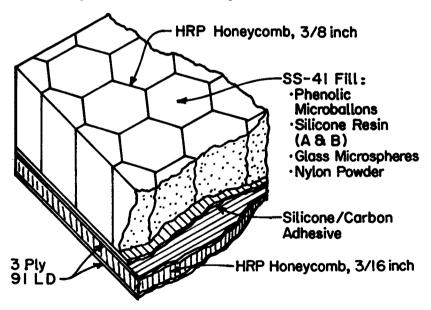


Figure 2 - Baseline Configuration - SS-41

Defects	Norma1	Varia	nces	Units
Defects	MOTIMAT	A	В	Units
Undercure, Overcure	250	150	350	o <sub>F</sub>
Net Density Variances	16	15	17	pcf
Filler Moisture Contents	0	5	10	%
Undermixing	45	15	30	Min.
Weak Bond to Honeycomb	45 minutes at 150°F	90 minutes	250 <sup>0</sup> F	In.
Horizontal Cracks in Fill	0	.25	.50	In.
Undercut Core	0	.10	. 20	ln.
Disbonds from Subpanel	0	1.0	1.5	
Broken Node Bonds	0	Center	Edge	
Broken Ribbons	0	Center	Edge	

Figure 3 - Defects Description, Final Testing

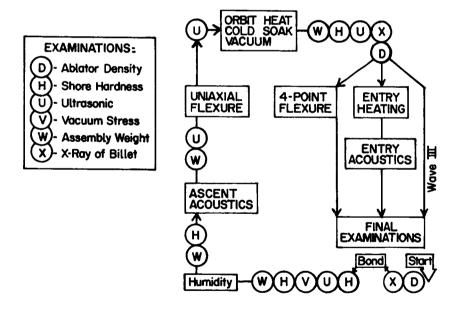


Figure 4 - Sequence of Environments and Examinations, Final Testing

		Γ	Den-	<u> </u>	Wet		Under	er	<u>-</u>		HH	15	Undercut		Dis-	-		_		
Environment Defect	Cure	e e	sity		Basics	82	Mix	_	Staging		Cracks	8	Core	_	Bonds	_	Nodes	-	Ribbons	ns
	A	В	A	В	A	В	A	$\overline{}$	A	-	Ą		V	H	A	B	A	B 4	A	В
Ascent Acoustics																$\dashv$				
Uniaxial Flexure																	-			
Thermal Vacuum			P7							P5										
Four Point Flexure	P1 P2		P1	P2	P2	P2	P1	Pl	P2	P1	P2	P2 I	P1 I	P1						
Reentry Heating		E1								E3		H	E2 H	E2 E3						
Descent Acoustics	E2 E6					<u>Е</u> 6			E6	E2 E3			E2 E	E2 E3						
Cross Sectioning	E5	E5	E5 E7						E5	E7										
CRITICAL DEFECTS	4		4			i	·		4	4		4	•	4						
Pre-Entry								퉵	Entry											
Pl Low Strength P2 High Modulus P5 Fill Separation P7 Material Shrinkage	Mod Sep Sep	ngth ulus arat Shr	i fon finka	90 90					E E E E E E E E E E E E E E E E E E E		ligh fater Jurfa Inter	High Backface Tempe Material Losses Surface Roughening Internal Char Crack Loose Cell Surfaces	face Lossioughi Char 1 Sui	Temes ening Cra	High Backface Temperature Material Losses Surface Roughening Internal Char Cracking Loose Cell Surfaces	s cure				
Definite									E7		)eep	Deep Pyrolysis	lysi	σ <sub>2</sub>						
Possible			<b>[34</b>	1gur	٠	. Sun	mary	of.	Crit	ical	Con	Figure 5 - Summary of Critical Comments on Defects	g on	Def	ects					